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TECHNICAL NOTE

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GAS CHROMATOGRAPHIC MEASUREMENT OF TRACE CONTAMINANTS IN A SIMULATED SPACE CABIN

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SUMMARY

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Methods have been developed for sampling the air in a simulated space cabin and analyzing it for trace contaminants by gas chromatography. Compounds which were identified during five simulated flights include acetaldehyde, acetone, methanol, ethanol, Freon-11, Freon-12, Freon-22, methyl chloride, and dimethyl sulfide. The presence of Freon-114, isoprene, acetylene, ethylene oxide, carbon tetrachloride, methyl ethyl ketone, benzene, and toluene is also suspected, although identity has not been confirmed unequivocally. Several additional unidentified compounds were also present. A number of unidentified peaks appeared transiently during the malfunction of a waste disposal unit. Thus, gas chromatography has great potential value for monitoring the atmosphere within space capsules for metabolic products as well as for other contaminants introduced accidentally.

Chemical tests were made for the presence of inorganic compounds in the atmosphere during two additional simulated flights. The presence of sulfate ion, chloride ion, chlorine, sulfur dioxide, ammonia, and ozone in trace amounts was confirmed.

INTRODUCTION

The maintenance of men in space for long periods of time in an enclosed cabin in which foods must be prepared and waste materials recycled creates new problems in air and water pollution that must be solved before prolonged flights are possible. The use of large conditioning units would be impractical because of the increase in payload. Therefore, means must be found to keep trace contaminants in the atmosphere below toxic levels with equipment having the least weight possible. Moreover, a means must be available to detect malfunction of this equipment, and the contamination of the atmosphere from this and other sources.

Contamination of the atmosphere in an enclosed cabin arises from normal metabolic processes such as breathing and perspiring. It also may arise from the preparation of food, the malfunction of equipment, or the presence of materials from which the emission of vapors is unsuspected. Therefore, careful control and monitoring of the atmosphere within a space cabin is essential.

Before optimum methods can be found to keep the air and water in a condition where they can be used with comfort and safety, detailed knowledge must be available on the nature of the compounds that are present, and the rates at which they are likely to be evolved. This should include both normal and stress conditions. For example, illness of personnel, the opening of a container of spoiled food, failure of a waste disposal unit, or fire from the short-circuiting of electrical equipment might produce temporary conditions that would overload the air purifier. Therefore, a study should be made of the types of compounds that are produced and how these are best removed from the atmosphere of a space cabin.

Gas chromatography is without doubt the most sensitive and versatile technique currently available for making such a study. Not only can it be used to detect trace contaminants at the parts per billion level, but it can also separate complex mixtures and provide a clue to the identity of each component. It is fast and requires relatively uncomplicated equipment. This technique, together with the proximity of a Space Cabin Simulator at the School of Aerospace Medicine in San

Antonio has made it possible to obtain a rapid insight into some of the problems which must be faced.

This report summarizes work carried out in this area during a contract between the National Aeronautics and Space Administration and the Southwest Research Institute.

PREVIOUS WORK DONE ON SUBMARINES

Most of the information previously available on trace contaminants in the atmospheres of enclosed habitable systems has been derived from experience with submarines. Originally, submarines were designed to submerge for short periods of time only, so removal of trace contaminants in the air was unnecessary. However, the advent of nuclear power has made it possible to build ships which can remain submerged for many weeks at a time. During this time, activated charcoal is used to adsorb organic compounds, and hydrogen and carbon monoxide are oxidized by catalytic combustion. The catalytic (Hopcalite) combustion unit is also capable of burning hydrocarbons when operated at a temperature of 350°C.

Many of the contaminants found in the atmospheres of submarines are characteristic of the particular type of operation involved, while others would be found in all types of enclosed habitable systems. In older submarines, hydrogen was produced during recharging of batteries. Although not toxic, it had to be removed from the air to avoid explosion hazards. An analysis of the materials desorbed from the charcoal air filters on the nuclear submarines Nautilus and Seawolf revealed that hydrocarbons were an important contaminant (Nestler, 1960). Analysis by mass spectrometry revealed that aliphatic paraffins, cycloparaffins, mono-olefins, cyclo-olefins, diolefins, acetylenes, and aromatics were present. In view of the similarity in composition between all samples, it is probable that relatively few sources account for a major part of the pollution observed in the nuclear ships. These include solvents used in paints and cleaning solutions. Other contaminants have been shown to be the Freons used in aerosols and refrigeration units. Although not toxic by themselves, they create a hazard when catalytic combustion units are used to purify the air, since HF and HCl are formed. Obviously, the foregoing

contaminants can be reduced or eliminated by using water-base paints and taking other precautionary measures.

The accumulation of hydrocarbons in the air not only presents a potential toxic hazard, but also places an added strain on the oxygen regeneration system. Thus 2.1 ppm of hydrocarbon in the air will consume as much oxygen as one man when the air is passed through a catalytic combustion chamber at a rate of 500 cfm (Johnson and Carhart, 1958).

Cigarette smoking also represents a source of air pollution for which control is optional. If smoking was permitted during periods of long submersion, it was essential to provide a method for removing carbon monoxide from the air formed by the incomplete combustion of tobacco (Thomas, 1960). A Hopcalite combustion unit was employed for this purpose, but, when operated at low temperatures, hydrocarbons interfered with its operation.

Many organic compounds are found in cigarette smoke and must be presumed to be present in the atmosphere of any enclosed system where smoking is permitted (Table I). These include alkanes, olefins, alcohols, carbonyl compounds, and even such compounds as methyl chloride. Moreover, interactions between these compounds and other contaminants in the air may create secondary problems in air pollution.

Certain contaminants which will occur in closed systems cannot be avoided, although they may be reduced by diet control and similar measures. These include odors from human wastes, perspiration, and foods. Hydrogen, methane, and hydrogen sulfide are present in flatus, which is discharged at a rate of 2 liters per day per person. Obviously, these problems are common to submarine and space travel.

A fairly large number of compounds have already been identified and measured quantitatively in submarine atmospheres (Table II). These include inorganic compounds such as ammonia, nitrous oxide, nitrogen dioxide, etc., as well as organic compounds such as methanol and methane. Others have been detected in the atmospheres but not measured (Table III). Some of these such as monoethanolamine and Freon obviously have been introduced with equipment, while others may be of biological origin. It is probable that the former will vary

TABLE I. SOME GASES AND VAPORS IDENTIFIED IN CIGARETTE
SMOKE AND PRESUMED TO EXIST IN
SUBMARINE ATMOSPHERES
(Piatt, 1960)

Material	Chemical Formula	Maximum Acceptable Concentration in ppm (ACGIH)
Acetylene	C_2H_2	-
Acetaldehyde	CH_3CHO	200
Acetone	CH_3COCH_3	1000
Benzene	C_6H_6	25
Butadiene	$CH_2:C:CHCH_3$	1000
Carbon Dioxide	CO_2	5000
Carbon Monoxide	CO	100
Ethane	C_2H_6	-
Ethylene	C_2H_4	-
Diacetyl	$CH_3COCOCH_3$	-
Furan	C_4H_4O	-
Isoprene	$CH_2:C(CH_3)CH:CH_2$	-
Hydrogen Cyanide	HCN	10
Isobutylene	$(CH_3)_2CCH_2$	-
Methane	CH_4	-
Methyl Alcohol	CH_3OH	200
2-Methyl Furan	$C_4H_3OCH_3$	-
Methyl Chloride	CH_3Cl	100
Propylene	C_3H_6	-
Methyl Ethyl Ketone	$CH_3COCH_2CH_3$	-
Furfuraldehyde	C_4H_3OCHO	5
Formaldehyde	$HCHO$	5
Propionaldehyde	C_2H_5CHO	-
Diethyl Ketone	$C_2H_5COC_2H_5$	-

TABLE II. COMPOUNDS QUANTITATIVELY IDENTIFIED IN SUBMARINE ATMOSPHERES
(Piatt, 1960)

Material	Chemical Formula	Type of Submarine (F = Fleet; N = Nuclear)	Highest Concentration Normally Found	Maximum Acceptable Concentration in ppm (ACGIH)
Acetylene	C ₂ H ₂	N	0.5 ppm	-
Ammonia	NH ₃	N	>1 ppm	100
Carbon Dioxide	CO ₂	F, N	1.1%	5000
Carbon Monoxide	CO	F, N	38 ppm	100
Chlorine	Cl ₂	F, N	1 ppm	1
Freon-12	CCl ₂ F ₂	F, N	70 ppm	1000
"Hydrocarbons" (other than CH ₄)	"HC"	F, N	25 ppm	-
Hydrogen Fluoride	HF	N	0.3 ppm	3
Hydrogen	H ₂	F, N	1.75%	-
Methane	CH ₄	F, N	118 ppm	-
Methyl Alcohol	CH ₃ OH	N	6 ppm	200
Monoethanolamine	HOCH ₂ CH ₂ NH ₂	N	<1 ppm	-
Nitrogen	N ₂	F, N	80%	-
Nitrogen Dioxide	NO ₂	N	0.1 ppm	5
Nitrous Oxide	N ₂ O	N	27 ppm	-
Oxygen	O ₂	F, N	20%	-
Stibine	SbH ₃	F, N	1 ppm	0.1
Water Vapor	H ₂ O	F, N	60% R. H.	-
"Cigarette Smoke"		F, N	0.4 µg/l	-

TABLE III. COMPOUNDS QUALITATIVELY IDENTIFIED IN TRACE AMOUNTS
IN SUBMARINE ATMOSPHERES
(Piatt, 1960)

Material	Chemical Formula	Type of Submarine (F = Fleet; N = Nuclear)	Maximum Acceptable Concentration in ppm (ACGIH)
Arsine	AsH ₃	F	-
Benzene	C ₆ H ₆	N	25
1-3-Dimethyl-5-ethylbenzene	1, 3-(CH ₃) ₂ -5-C ₂ H ₅ C ₆ H ₃	N	-
Ethylene	C ₂ H ₄	N	-
p-Ethyl Toluene	1, 4-CH ₃ C ₂ H ₅ C ₆ H ₄	N	-
Freon-114	CF ₂ ClCF ₂ Cl	N	1000
"Gasoline Vapors"	-	F	500
Hydrogen Chloride	HCl	N	5
Mesitylene	1, 3, 5-(CH ₃) ₃ C ₆ H ₃	N	-
Propane	C ₃ H ₈	N	-
Pseudocumene	1, 2, 4-(CH ₃) ₃ C ₆ H ₃	N	-
Sulfur Dioxide	SO ₂	F, N	5
Toluene	C ₆ H ₅ CH ₃	N	200
o-Xylene	1, 2-(CH ₃) ₂ C ₆ H ₄	N	200
m-Xylene	1, 3-(CH ₃) ₂ C ₆ H ₄	N	200
p-Xylene	1, 4-(CH ₃) ₂ C ₆ H ₄	N	200

with the type of operation, while the latter should occur more or less uniformly in all inhabited enclosed systems. Therefore, it is of interest to note that the compounds found in the atmospheres of submarines do not parallel those found in the space capsule at the School of Aerospace Medicine (Table IV). This may be due to differences in experimental techniques. In the submarine experiments, the contaminants were desorbed from the charcoal used in the air filters by vacuum extraction or steam distillation, while, in the experiments carried out at Southwest Research Institute, contaminants were trapped directly from the atmosphere. It seems likely that some of the compounds of biological origin may have been lost in the submarine experiments.

EXPERIMENTAL METHODS

Space Cabin Simulator

The space cabin simulator at the School of Aerospace Medicine, located at Brooks Air Force Base, San Antonio, Texas, was used for these studies. Figure 1 is a cutaway drawing of the simulator which is designed to study the physiological and psychological stresses on man during prolonged periods of isolation. Space is provided to accommodate two men for periods up to 30 days in complete isolation. The crew members can be observed through observation ports or with remote TV circuits. Equipment is provided for monitoring of physiological functions. The simulator is equipped with an environmental control system, simulated navigational controls to test crew responses, a bed, sanitary facilities, food storage areas, and cooking facilities.

The environmental control system is of special interest in connection with the study of atmospheric contaminants. Baralyme $[\text{Ba}(\text{OH})_2 \cdot \text{Ca}(\text{OH})_2 \cdot 8\text{H}_2\text{O}]$ is used to absorb carbon dioxide, and oxygen is provided from high pressure tanks. The atmospheric pressure can be controlled at any desired value between sea level and 28,000 feet equivalent. Frequently, one-half atmosphere is used which is equivalent to approximately 18,000 feet. At this pressure, the atmospheric composition is usually maintained at 40% oxygen and 60% nitrogen, so that the partial pressure of oxygen is approximately

TABLE IV. COMPOUNDS IDENTIFIED OR TENTATIVELY
IDENTIFIED IN SPACE CABIN SIMULATOR

<u>Compounds Identified in Cabin Air by GLC</u>	<u>Max. Conc., (mg/m³ at 25°C and 745 mm)</u>	<u>Remarks</u>
Acetaldehyde ²	0.53	
Acetone ¹	2.05	
Methyl Alcohol ¹	0.58	
Ethyl Alcohol ¹	16.6	
Freon-12 ¹	185.0	Flight #2 only
Freon-11 ¹	47.0	Flight #2 only
Methyl Chloride ¹	2.3	Flights #1, #2 only
Freon-22 ¹	0.6	Flights #3, #4 only
Dimethyl Sulfide ²	0.012	
<u>Compounds Suspected in Cabin Air by GLC</u>		
Freon-114 ³	0.08	Flight #3 only
Isoprene ²	0.08	
Acetylene ⁴	0.18	Flights #3, #4 only
Ethylene Oxide ⁴	0.2	
Carbon Tetrachloride ⁴	0.035	
Methyl Ethyl Ketone	0.3	
Benzene ⁴	0.2	
Toluene ⁴	0.13	
Propionaldehyde	0.011	
Isobutyraldehyde	0.006	
Methyl Isobutyl Ketone	0.31	
Methyl Propyl Ketone	0.40	
Unknown A	0.57	Flight #2 only
Unknown B	5.3	Flight #2 only
<u>Materials Detected by Chemical Tests</u>		Flights #6, #7 only
Sulfate ion	1.95	
Chloride ion	1.80	
Sulfur dioxide	0.148	
Chlorine	0.088	
Ammonia	1.86	
Ozone	0.025	

Identification

1. Infared spectra of collected fraction
2. Odor + retention time
3. Halide detector + retention time
4. Retention times on two different columns

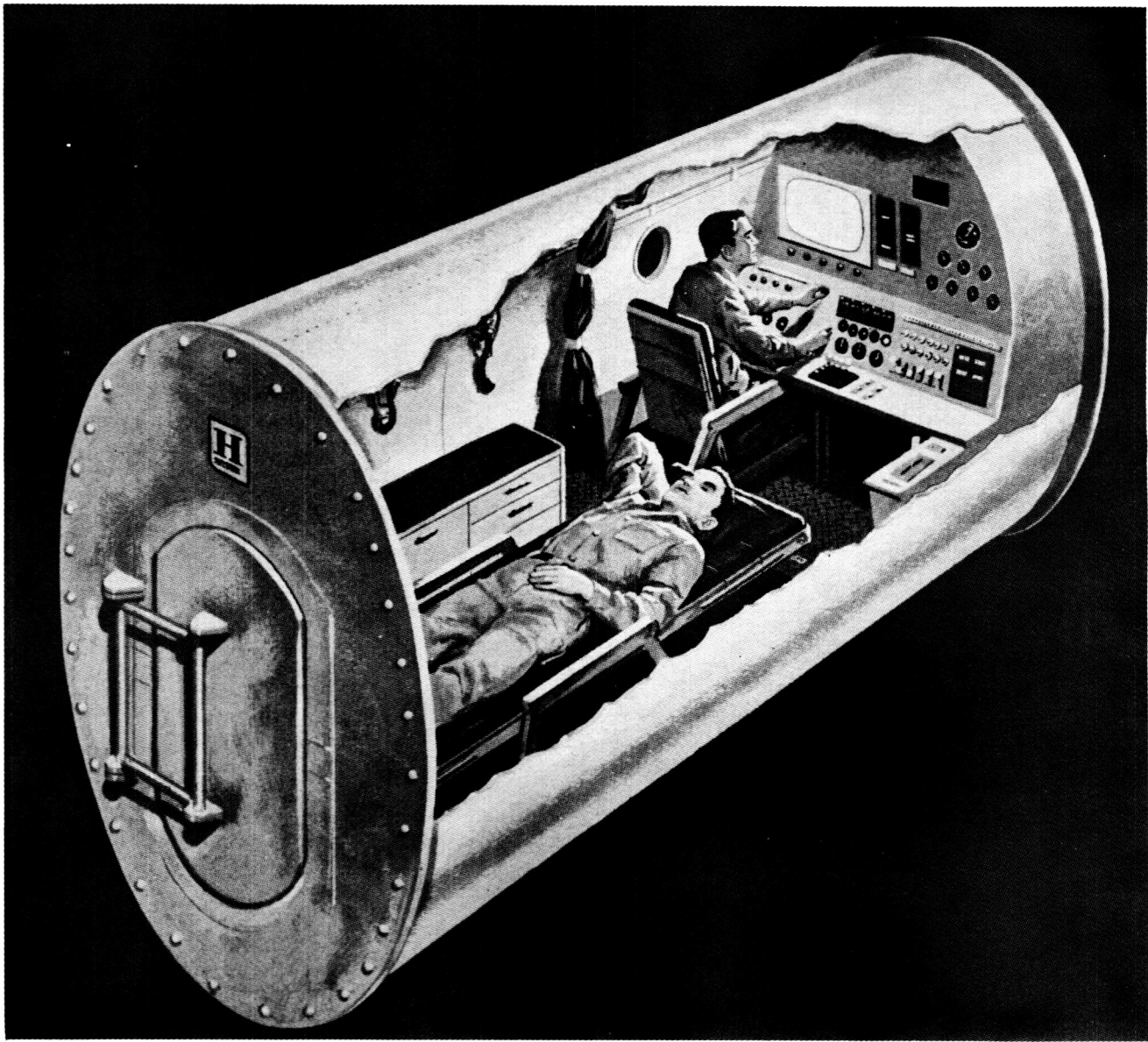


FIGURE 1. CUTAWAY VIEW OF SPACE CABIN SIMULATOR

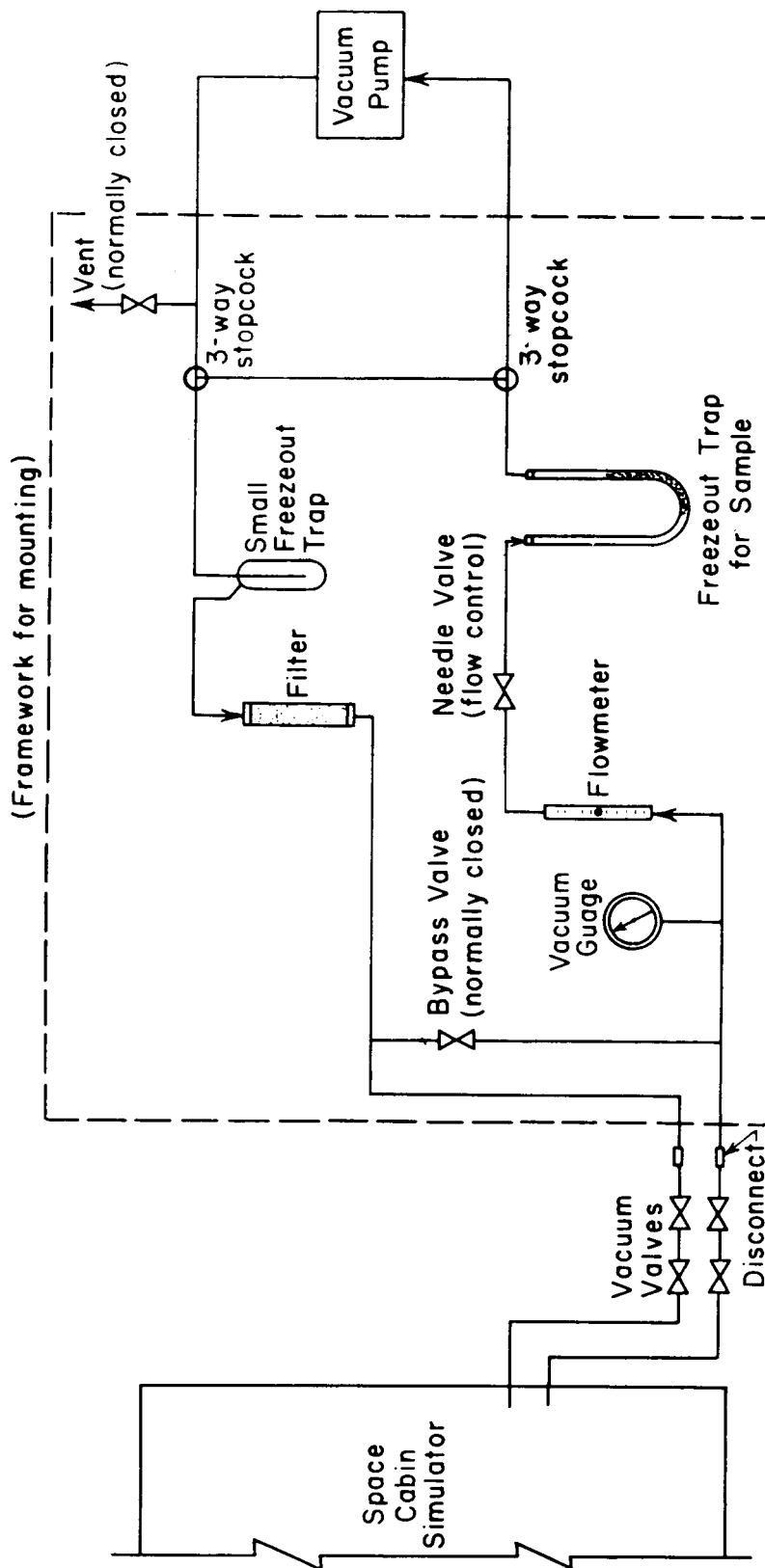
that in a normal atmosphere. Temperature and humidity also can be controlled at any desired level, and an electrostatic precipitator is provided to remove dust and smoke particles from the air being recirculated. At a later date, a photosynthetic gas exchanger can be installed, thus duplicating all the environmental aspects of a space ship with a regenerative atmospheric control system.

The major difference between this simulator and a probable space ship is that the environmental control system was designed without much regard for weight limitations. Approximately 50 pounds of activated carbon is provided for air purification in a series of large canisters, and the entire volume of air in the simulator is recycled once a minute. This could mean that trace contaminants in the simulator are removed more rapidly and completely than would be the case in an actual space ship. Therefore, future work should include experiments in which fewer canisters or a slower rate of recycling is used.

Sampling Air from the Space Cabin Simulator

Figure 2 shows a schematic diagram of the equipment used for sampling air from the space cabin simulator. Figure 3 shows a photograph of this equipment mounted on a portable framework in such a way that it can be used adjacent to the cabin at any time and moved readily from place to place. Two stainless steel 1/4-inch sampling lines were mounted in the end of the cabin, each provided with two stainless steel high vacuum valves in series. The valves were used in series to insure that no leakage would occur to upset the environmental control system in the space cabin simulator. Each line can be connected to the corresponding line on the sampling equipment with a 3/4-inch length of tygon tubing, and then disconnected after the sample has been taken. Except for this short length of tubing, the air being sampled comes into contact with stainless steel and glass only until after it has passed through the freezeout trap where impurities are collected. A short tube containing Ascarite is placed between the inlet and the sample collector to remove carbon dioxide. Otherwise, more of this gas condenses in the trap than can be handled by the system used for analysis.

To collect a sample, the entire sampling system is evacuated. The valves in the sampling line are opened to connect the sampling



Vacuum Gauge, Duragauge, 0-30 in. Vacuum.
Flowmeter, Tri-Flat No 9144, 0-2 liters/min.
Vacuum Pump, Welch Duo-Seal, Single Stage.
Vacuum Valves, Hoke No 413, Stainless Steel
with Monel Diaphragm.

Vent, Bypass, and 3-way Stopcocks provided

to evacuate entire system before opening
valves into Space Cabin Simulator.

Small Freezeout Trap and Filter (Carbon
and Silica Gel) provided to remove oil
and dirt from Vacuum Pump, before
returning air to Space Cabin Simulator

FIGURE 2. SCHEMATIC DIAGRAM OF EQUIPMENT USED TO SAMPLE THE
ATMOSPHERE IN THE SPACE CABIN SIMULATOR

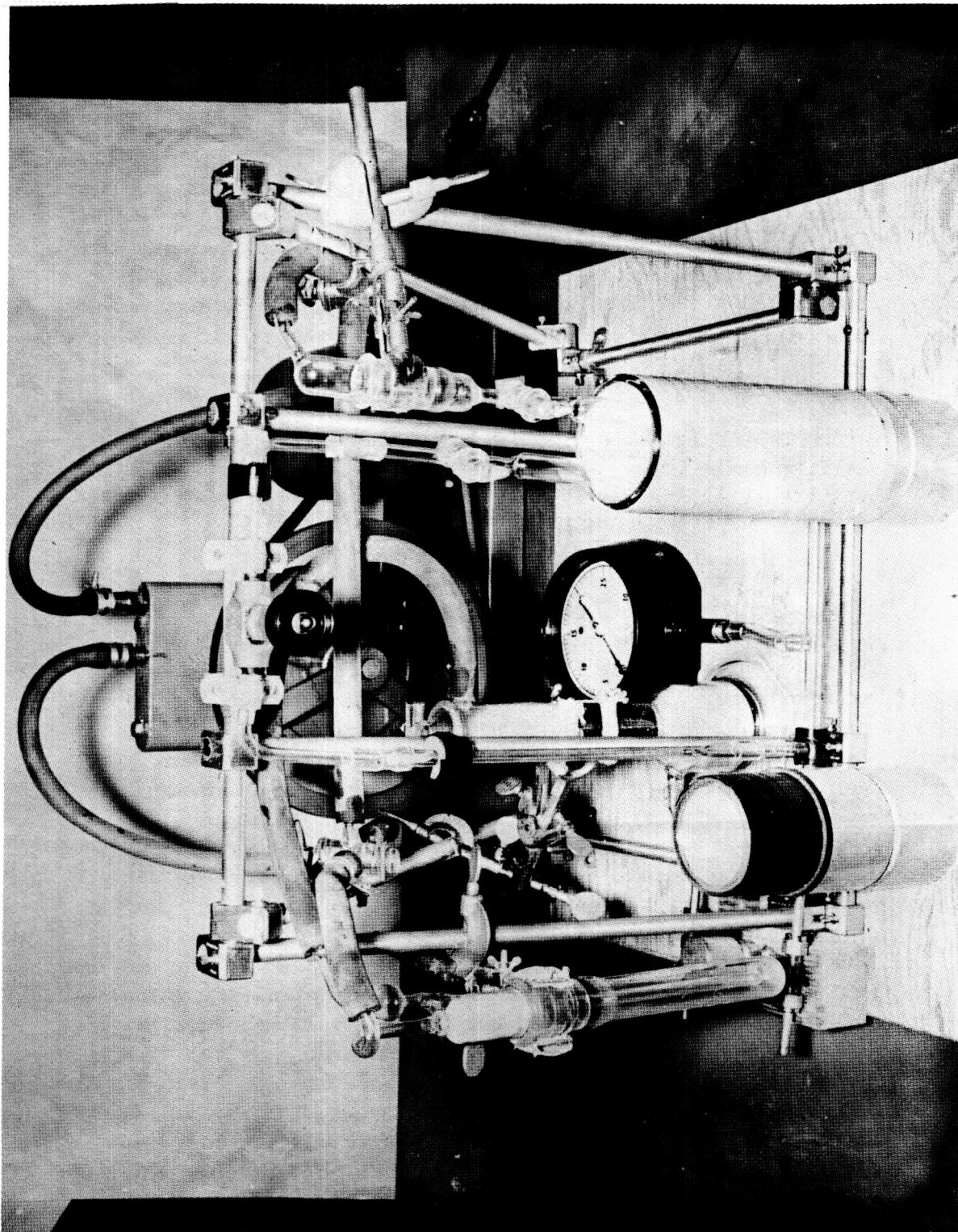


FIGURE 3. PHOTOGRAPH OF EQUIPMENT USED TO SAMPLE THE AIR
IN THE SPACE CABIN SIMULATOR

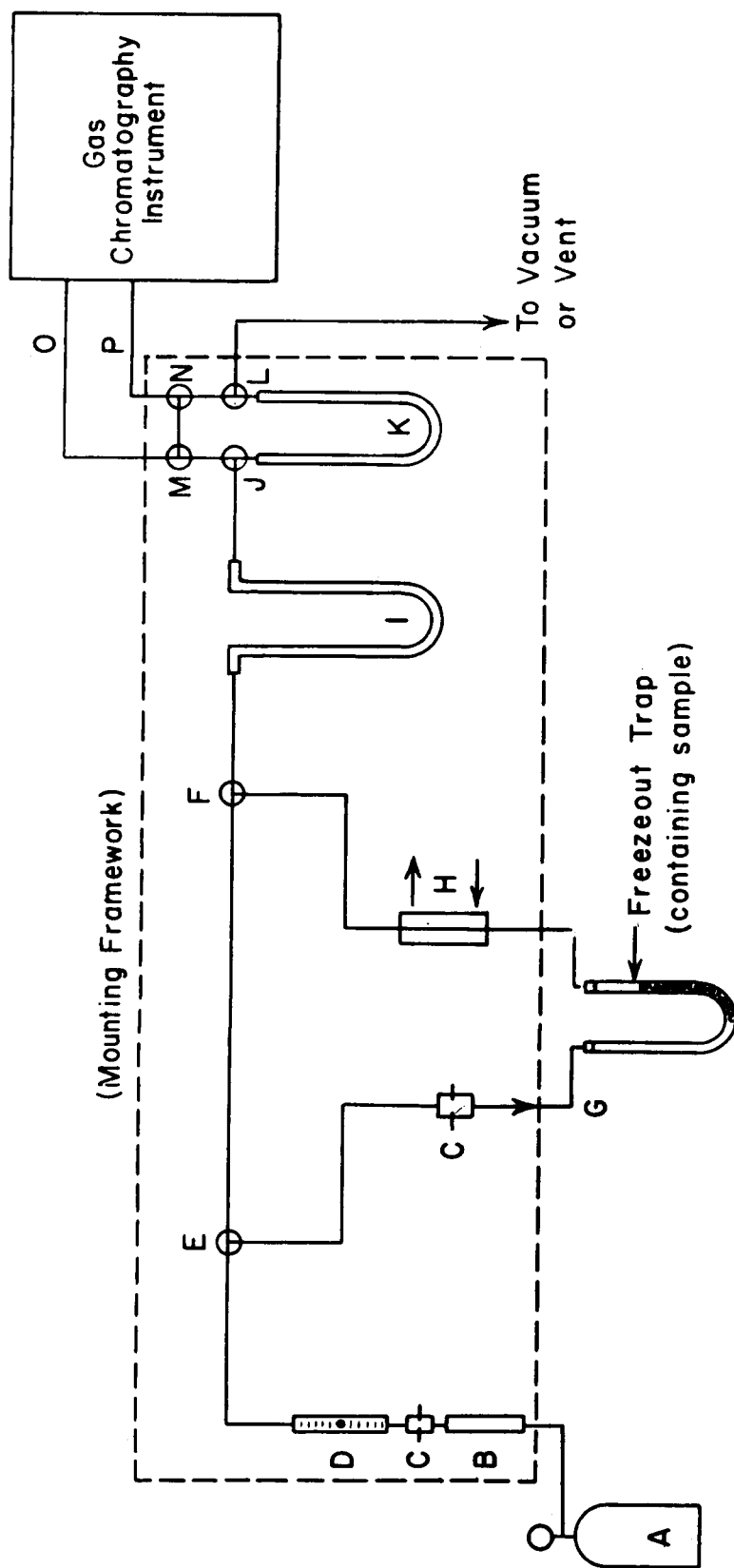
system to the atmosphere of the space cabin simulator. Air is then passed from the simulator through a rotameter and freezeout trap, through the vacuum pump, through a filter and cold trap to remove any oil picked up in the vacuum pump, and finally back into the simulator. The freezeout trap is made from a U tube of 15-mm Pyrex tubing, with Pyrex wool in the outlet side. The inlet side is left open so that ice (from water vapor) can collect near the inlet without plugging the trap. Similar apparatus has been used for collecting samples in air pollution surveys, and it is known that such traps will retain quantitatively practically any volatile material which can be condensed at the operating temperature. The use of liquid nitrogen at a cabin pressure of 0.5 atmosphere is satisfactory, since oxygen is not condensed under these conditions.

The leak rate of the apparatus was found to be 1.3 ml of free air per hour when operated at 0.5 atmosphere. The leak rate of the simulator itself is about 1 liter per hour at this pressure, so leakage into the sampling system is negligible by comparison.

Analysis of Samples

Samples were analyzed by gas-liquid chromatography using Carbowax or Silicone 550 as the stationary phase. Before introducing the volatiles into the instrument, it was necessary to remove condensed water vapor by the apparatus shown in Figure 4. Normally, this equipment is used to remove carbon dioxide also. However, the amount present in these samples was so large that it exceeded the capacity of the apparatus to handle it. Therefore, carbon dioxide was absorbed by Ascarite before the volatiles were condensed, as described previously.

The apparatus is designed so the freezeout trap can be disconnected from the sampling device and mounted in the analytical train at point G. It is then evacuated to remove organic volatiles or other materials from the bulk of the water. The stripping is aided by a small flow of helium from tank A which passes through a drying tower B, a flow restrictor C, a flowmeter D, and sweeps the sample out of the freezeout trap which is maintained at approximately room temperature. The helium carrying the sample then goes through a



- | | |
|--|--|
| A - Helium supply | E, F, J, L, M, N - 3-way stopcocks |
| B - Drying tube | I - Preliminary column (7 mm tubing x 10 inches packed with Carbowax 1500-C-22 firebrick) |
| C - Capillary to limit flow | K - Freezeout trap (packed with glass beads) |
| D - Flowmeter | O - Helium carrier gas line from reference side of detector |
| G - Helium inlet for sampling device | P - Connection to chromatographic column |
| H - Helium return from sampling device, through ice water condenser | |

FIGURE 4. SCHEMATIC DIAGRAM OF APPARATUS USED TO SEPARATE TRACE COMPONENTS FROM WATER PRIOR TO GAS-LIQUID CHROMATOGRAPHY

condenser H to remove part of the water, and then to the precolumn I which is immersed in liquid nitrogen. The remaining water plus the volatile constituents to be analyzed are frozen at the inlet to the precolumn and are retained there until sweeping is complete. The precolumn is then heated to 60°C and helium swept through it to separate the volatiles from the remaining water. Helium is swept through the column, and the volatiles are re-collected in the liquid nitrogen trap K. The water is then vented to the atmosphere. Next, trap K is warmed, and the volatiles are swept into the chromatograph for analysis.

Conditions for Chromatography

Chromatograph	Perkin-Elmer Model 154A or 154D Vapor Fractometer equipped with 0.75 m × 0.6 cm o. d. precolumn packed with Carbowax 1540 and Polyglycol E 300 (50:50 mixture on 35/48 Chromosorb P (15:85) (Rhoades, 1958).
Column dimensions	2-meter × 0.6 cm o. d. glass U tube.
Solid support	I & II. Untreated Chromosorb P (F&M Scientific Co.) (35/40 U.S. mesh). III. Same as I & II but 35/48 U.S. mesh.
Stationary phase	I. Carbowax 1540 (45%) and Polyglycol E 300 (55%) (15:85). II. Dow Corning 550 Silicone (20:80). III. Carbowax 1500 (15%) and Dow Corning 200 (10%) (25:75).
Temperature	40°C throughout.
Carrier gas	Helium at 60 ml/min at outlet. Inlet pressure: 6 psig. Outlet pressure: atmospheric.
Recorder	Minneapolis Honeywell or Leeds and Northrup. 1 mv; 1 second; 20 inches/hour.

Detector	Thermistor operated at 5.6 v (154A) or 8.0 v (154D).
Sample size	Volatiles trapped from 20 liters of air at STP.
Analysis time	I. 1.8 minutes to acetone. II. 2.5 minutes to acetone. III. 6.5 minutes to acetone.

Analysis of Inorganic Contaminants

For the analysis of sulfate, chloride, and ammonium ions, 7 cubic feet of cabin air at atmospheric pressure was bubbled through 40 ml of distilled water at a rate of 1/4 cfm. Aliquots of this solution were then analyzed as follows:

Sulfate ion. A salt-acid solution was prepared containing NaCl (240 g) and conc. HCl (20 ml) diluted to one liter with distilled water. One ml of this solution was added to a 10-ml aliquot of the sample collected above. Crystals (15) of barium chloride were then added, and the transmittance of the resulting suspension was read on a colorimeter at a wave length of 525 mμ using pure water plus the reagents as a blank. (Am. Pub. Health Assn., 1955.)

Chloride ion. Silver nitrate crystals (0.1 g) and 1:1 nitric acid (1 ml) were added to a 10-ml aliquot of the sample. The transmittance of the solution was read on a colorimeter at 525 mμ using a reagent blank. (Scott, 1939.)

Ammonium ion. Nessler's reagent (1 ml) was added to a 10-ml aliquot of the sample. After 15 minutes, the absorbance was read at 410 mμ against a reagent blank. (Am. Pub. Health Assn., 1955.)

Sulfur dioxide and chlorine were measured on separate samples taken by bubbling cabin air through solutions of reagents. These analyses were made as follows:

Sulfur dioxide. Cabin air (2 ft^3) was bubbled through $0.1\text{ M Na}_2\text{HgCl}_4$. This solution was then treated with 0.04% p-rosaniline hydrochloride (1 ml) and 0.2% formaldehyde (1 ml). After 20 minutes, the absorbance was read on a colorimeter at $560\text{ m}\mu$. (West and Gaeke, 1956.)

Chlorine. Cabin air (2 ft^3) was bubbled through 0.1 N NaOH (10 ml). o-Tolidine reagent (1 ml) and 1:1 sulfuric acid (1 drop) were then added and the absorbance read at $445\text{ m}\mu$. (Am. Pub. Health Assn., 1955.)

Ozone. Cabin air (2 ft^3) was pulled through a gas scrubber containing 20% KI solution (10 ml). A portion of the solution was then transferred to a colorimeter tube and the absorbance measured at $335\text{ m}\mu$ against a reagent blank. (Littman, 1953.)

RESULTS

Identity of Volatile Compounds Present in Cabin Air

The volatile compounds present in the air within the simulator yield chromatograms with characteristic retention times (Fig. 5). However, the same compounds were not found in all experiments owing to equipment changes and the fact that chromatographic columns having different resolving power were sometimes used.

Compounds that were identified with certainty include acetaldehyde, acetone, methanol, ethanol, Freon-12, Freon-11, methyl chloride, Freon-22 and dimethyl sulfide (Table IV). Compounds which are suspected to be present include Freon-114, isoprene, acetylene, ethylene oxide, carbon tetrachloride, methyl ethyl ketone, propionaldehyde, isobutyraldehyde, methyl isobutyl ketone, methyl propyl ketone, benzene, and toluene. Other peaks appeared from time to time whose identity is entirely unknown. In addition, the presence of chloride ion, sulfate ion, chlorine, sulfur dioxide, ammonia, and ozone in trace amounts was indicated by chemical tests.

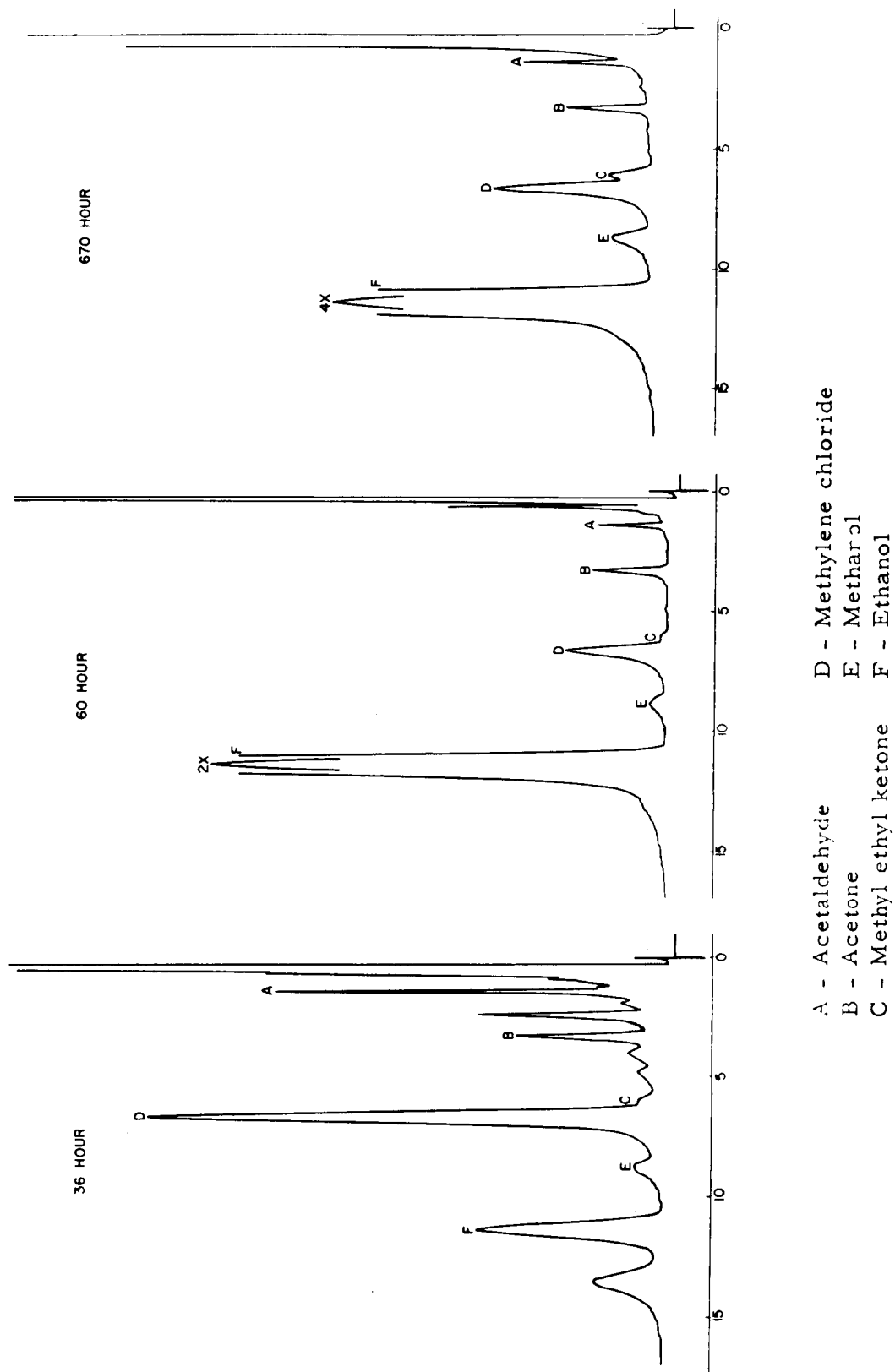


FIGURE 5. CHROMATOGRAM OF VOLATILES FOUND IN THE ATMOSPHERE IN THE FIRST SIMULATED SPACE FLIGHT AT 36, 60, AND 670 HOURS AFTER THE BEGINNING OF THE EXPERIMENT

The contaminants in the cabin air were found to be present at an extremely low level, so collection for identification by infrared analysis was not practical. However, larger amounts of them could be obtained by eluting the charcoal used to purify the cabin air.

The adsorbed materials were released from 50 grams of charcoal by wetting it and stripping it with an inert gas at reduced pressure, the volatiles being collected in a short chromatographic column immersed in liquid nitrogen. This column served to separate the released compounds from the water carried over during the stripping operation. The water-free contaminants were then re-collected and introduced into the analytical column for separation. The individual components were collected separately in cold traps.

Although the sizes of the peaks representing the volatiles collected from the charcoal filter were much greater than found in cabin air, the retention times were the same. Therefore, it was assumed that the contaminants recovered from the charcoal were the same as had existed in the cabin air.

The material collected in the traps was warmed and passed into an evacuated infrared cell and the spectra of the vapors recorded. The spectra were then compared to those of known compounds. The following compounds were positively identified: acetone, methylene chloride, methanol, and ethanol.

The substance believed to be acetaldehyde was not recovered in large enough amounts to obtain an infrared spectrum on it. However, this is not surprising, since it is a reactive compound and probably underwent aldol condensation when collected on the charcoal. The identity of methyl ethyl ketone was not confirmed either, but it seems likely that the tentative identification based on retention time is correct, since this compound (as well as acetaldehyde) has previously been identified in breath samples.

A number of smaller peaks were observed on the chromatogram obtained from the charcoal sample, but the components were not present in large enough amounts for infrared analysis.

Time Studies on Cabin Air

First Cabin Experiment. At approximately 10:30 p.m. on August 15, 1960, two Air Force officers commenced a 30-day simulated space flight in the Space Cabin Simulator.

The test was conducted at a total pressure of 380 mm Hg in an atmosphere containing 40% oxygen and 60% nitrogen. The carbon dioxide content (as measured by cabin monitoring instruments) on occasion was as high as 14 mm, but the usual range was 5 to 8 mm. The carbon monoxide content became excessively high on the 19th of August, and the cabin was "flushed" twice. "Flushing" was accomplished by raising the cabin pressure from 380 mm to 450 mm and then pumping out cabin air until the pressure was again reduced to 380 mm. Flushing was also required once each on the 21st of August, the 24th of August and the 2nd of September. "Flushing" was required because carbon monoxide was expelled into the cabin air by a malfunction of the waste disposal combustion unit. This unit apparently had been assembled incorrectly, and the off gases failed to pass through the Hopcalite combustion unit.

The air circulating system used in this test contained 525 pounds of Baralyme for carbon dioxide removal and 50 pounds of activated charcoal for air purification. A complete turnover of air was maintained once every minute in the cabin. A small portion of this was passed through Hopcalite for carbon monoxide removal.

Twenty-liter air samples were taken for analysis according to the method given under Experimental Methods. Samples were collected at midmorning and were analyzed 2 to 3 hours later. At the time of sampling, the concentrations of carbon monoxide and carbon dioxide were obtained from the cabin monitoring instruments. Samples were obtained daily for the first two weeks and then twice a week thereafter until the conclusion of the test.

The results of the analyses are given in Table V. Data are given only for peaks which appeared with regularity from sample to sample. Occasionally small additional peaks would appear in one sample, but would not be present in the following sample. These additional peaks are assumed to have been caused by some transient condition. Presumably these materials were removed by the charcoal filters with continued circulation.

TABLE V. IMPURITIES IN SPACE CABIN SIMULATOR IN TWO-MAN, 30-DAY TEST STARTED
ON 8/15/60 WITH AN ATMOSPHERE CONTAINING 40% O₂ AND 60% N₂
AT A PRESSURE OF 380 MM Hg

Compounds Measured	Amount Present at Hours (mg/m ³ at 25°C and 745 mm)														
	12	36	60	85	156	180	204	228	252	324	348	504	526	646	670
Acetaldehyde	0.03	0.38	0.08	0.24	0.14	0.07	0.14	0.11	0.09	0.12	0.08	0.22	0.10	0.08	0.11
Acetone	0.30	0.33	0.20	0.24	2.05	0.59	0.53	0.48	0.48	0.34	0.83	1.50	0.25	0.16	0.20
Methyl Ethyl Ketone	0.03	0.08	0.03	0.20	0.08	0.08	0.10	0.08	0.33	0.11	0.08	0.06	0.05	0.10	0.10
Methylene Chloride	0.15	2.30	0.46	0.90	1.13	0.98	0.81	0.60	0.86	0.75	0.81	2.00	1.60	1.40	0.79
Methanol	0.15	0.18	0.10	0.10	0.23	0.15	0.28	0.13	0.58	0.26	0.38	0.55	0.20	0.26	0.29
Ethanol	1.9	1.6	8.4	0.65	4.9	1.5	1.8	2.8	16.6	4.5	2.3	11	6.2	7.4	12
Unknown	0	0.60	0	0.05	0.10	0	0.10	0.09	0	0.10	0.05	0	0	0	0
CO (ppm)*	30	165	50	63	46	43	30	45	25	51	-	-	-	-	-
CO ₂ (mm Hg)*	3.9	6.0	4.4	5.4	6.3	5.5	7.9	6.2	8.5	8.0	7.8	14	3.8	-	4.0

*Data obtained from School of Aviation Medicine.

Chromatograms of the 36-, 60-, and 670-hour samples are shown in Figure 5. A number of peaks were recorded in the 36-hour sample that did not appear in the 60-hour sample. Shortly before the 36-hour sample was taken, there was a malfunction of the waste disposal unit, and it is assumed that the additional peaks were caused by decomposition products expelled into the cabin air as a result of this malfunction. The 60-hour sample did not show these additional peaks.

The data indicate that there was very little buildup of the compounds measured during the entire 30 days. The rather large amount of ethanol and the wide variation shown are due to the use of this material within the cabin by the test personnel for hand washing. The relatively high value for acetone obtained at 156 hours was due to the fact that a bottle of this solvent was opened within the cabin to repair a crack in a Plexiglas window.

For the most part, the materials measured appear to be well below the maximum allowable concentrations listed in Tables I to III.

However, it should be noted that some highly volatile materials were present that were eluted from the column too rapidly to be resolved. These were collected and identified in a second simulated space flight.

Second Cabin Experiment. At approximately 10:30 a.m. on 24 January 1961, two Air Force officers commenced a 17-day simulated space flight in the cabin at the School of Aerospace Medicine. The oxygen in the cabin was maintained at 180 mm Hg. Small amounts of water vapor, carbon dioxide, and nitrogen present in the cabin raised the total pressure to approximately 190 mm. The incinerator which had functioned poorly in the previous test was removed, and a water still of new design replaced the unit used in the first test. The air purification system was the same as used previously.

Sampling was carried out as described in Experimental Methods. The first two samples were analyzed on a Silicone 550 column in order to resolve the fast moving compounds observed in the first test. The remaining samples were chromatographed on Carbowax. The material which was eluted during the first 1-1/2 minutes was re-collected and chromatographed again on silicone. Two fractions were obtained. Infrared examination showed they were Freon-11 and Freon-12. The

concentrations of these materials increased throughout the test until they became abnormally high compared to those of other atmospheric contaminants. Finally, it was discovered that these gases were liberated into the cabin atmosphere through the use of an aerosol can containing an antifoam agent which was being added to the water recovery still. When the use of the aerosol was discontinued, the concentration of Freons dropped to a lower level for the remainder of the test. However, Freon-12 still remained the predominant atmospheric contaminant. Results are shown in Table VI.

Third Cabin Experiment. On March 14, 1961, two Air Force officers commenced a 30-day simulated space flight. As in previous tests, the air purification system consisted of beds of Baralyme and charcoal. The cabin atmosphere was held at 150 mm of oxygen and the total pressure at 0.5 atmosphere. Both cabin occupants were non-smokers.

In these experiments, a gas chromatographic packing containing 15% Carbowax 1500 and 10% D. C. 200 Silicone Fluid was used. This column, unlike the silicone column previously used, exhibited very little tailing of the more polar compounds, but still retained the ability of increasing the retention volumes of those compounds which were eluted very rapidly on Carbowax. A 2-meter column was used, and the solid support was 35/48 U. S. mesh Chromosorb P.

This column appears to be quite well suited for the analysis of the cabin air. The increased retention times enabled the analyses to be performed on a single column, and the improved resolution indicated the presence of additional compounds (Table VII). It is assumed that most of the additional compounds found were also present in the first two simulated tests but were undetected on the columns used at that time. The concentrations of these additional compounds were quite low and consistent, with the single exception of Freon-22, which showed a 10-fold increase during the last few days of the 30-day simulated test.

Methylene chloride also was found in the cabin atmosphere, but in lower concentrations than in the previous test. This was anticipated, since it was believed to have emanated from a plastic which had since been removed.

TABLE VI. IMPURITIES IN SPACE CABIN SIMULATOR IN TWO-MAN, 17-DAY TEST STARTED
ON 1/24/61 AT 180 MM Hg OF O₂ AND 190 MM TOTAL PRESSURE

Compounds Measured	Amount Present at Hours (mg/m ³ at 25°C and 745 mm)														
	0	24	48	72	144	168	192	216	240	264	312	336	360	384	
Freon-12	0.028	1.6	8.9	5.2	17	12	20	40	185	38	29	29	27	15	
Freon-11	2.6	0.84	4.4	1.8	7.8	8.8	5.1	16	47	13	5.1	4.1	3.4	1.9	
Acetaldehyde	*	*	0.11	0.26	0.28	0.31	0.37	0.42	0.50	0.44	0.53	0.53	0.42	0.19	
Acetone	*	*	0.41	0.84	0.31	0.37	0.48	0.43	0.54	0.52	0.44	0.60	0.46	0.25	
Methylene Chloride	*	*	0.15	0.13	0.12	0.11	0.15	0.16	0.15	0.18	0.11	0.33	0.12	0.07	
Unknown A	*	*	0.43	0.35	0.40	0.28	0.30	0.27	0.32	0.57	0.38	0.29	0.22	0.16	
Methanol	*	*	0.21	0.36	0.38	0.44	0.52	0.47	0.85	0.49	0.51	0.70	0.36	0.28	
Ethanol	*	*	2.9	4.2	3.0	3.4	4.3	4.1	5.3	4.5	4.2	3.3	3.6	2.0	
Unknown B	*	*	1.8	1.7	1.4	1.3	1.8	1.7	1.6	1.1	1.1	1.6	1.2	0.55	

*Sample analyzed on silicone column only.

TABLE VII. IMPURITIES IN SPACE CABIN SIMULATOR IN TWO-MAN, 30-DAY TEST STARTED ON 3/14/61 WITH 150 MM Hg OF O₂ AND 0.5 ATMOSPHERE TOTAL PRESSURE

Compounds Measured	Amount Present at Hours (mg/m ³ at 25°C and 745 mm)													
	24	72	144	196	240	312	360	408	480	528	576	648	696	
Acetylene	0.06	0.13	0.12	0.04	0.09	0.14	0.15	0.11	0.11	0.10	0.09	0.14	0.18	
Freon-22	0.003	0.002	0.006	0.008	0.012	0.002	0.003	0.003	0.003	0.003	0.004	0.46	0.60	
Freon-114	0.07	0.02	0.03	0.04	0.05	0.05	0.05	0.04	0.04	0.05	0.07	0.08	0.08	
Ethylene Oxide	0.12	0.09	0.23	0.18	0.21	0.07	0.06	0.07	0.07	0.07	0.07	0.07	0.06	
Acetaldehyde	0.01	0.02	0.03	0.03	0.02	0.02	0.03	0.04	0.06	0.06	0.06	0.06	0.06	
Isoprene	0.02	0.02	0.08	0.05	0.07	0.03	0.02	0.04	0.02	0.02	0.02	0.02	0.04	
Dimethyl Sulfide	0.006	0	0.006	0.006	0.01	0.006	0.006	0.009	0.006	0.006	0.006	0.006	0.006	
Acetone	0.14	0.28	0.48	0.45	0.69	0.46	0.47	0.35	0.76	0.38	0.51	0.46	0.27	
Carbon Tetra-chloride	0	0	0.03	0.03	0.04	0.02	0.02	0	0.01	0.01	0.02	0	0	
Methanol	0	0.08	0.12	0.10	0.20	0.05	0.07	0.08	0.11	0.06	0.11	0.06	0.10	
Benzene	0.19	0.13	0.21	0.11	0.11	0.09	0.08	0.15	0.09	0.06	0.06	0.12	0.12	
Methyl Isobutyl Ketone	0.16	0.16	0.36	0.40	0.45	0.07	0.16	0.16	0.18	0.18	0.18	0.22	0.18	
Toluene	0	0.07	0.08	0.08	0.13	0	0	0	0	0	0	0	0	
Ethanol	0.20	0.24	0.40	0.35	0.43	0.16	0.23	0.24	0.31	0.19	0.29	0.20	0.32	

Other compounds which were detected include acetone, acetaldehyde, methanol, and ethanol. In addition, two unknowns appeared in comparable concentrations that were designated as A and B. Efforts to identify them were unsuccessful. They were not present in the condensed sample in amounts large enough for infrared analysis. Furthermore, they were evidently bound irreversibly by the charcoal filter, since efforts to secure samples from this source failed. If it becomes necessary to identify the substances giving rise to these peaks, a larger air sample will have to be taken, or air circulation through the charcoal filter will have to be cut off for a period of time to allow the concentration of trace impurities to build up. This would probably be a worthwhile experiment in any event, since it would permit direct measurement of the total capacity a filtering system would have to have to be effective. Also, it would be a premeasure of what toxic effects might be anticipated if the air purification system failed in space.

Fourth and Fifth Cabin Experiments. These experiments were of 17 days duration, and the cabin contained two men. In both cases, the pressure was maintained at 180 mm Hg of O₂ and about 190 mm Hg total pressure. The results (Tables VIII and IX) are substantially equivalent to those obtained in the third experiment.

Sixth and Seventh Cabin Experiments. Since the composition of the organic trace components had not varied significantly in Experiments 3 to 5, inorganic compounds were measured in the following two experiments by conventional chemical methods. The results are shown in Tables X and XI. The presence of trace amounts of sulfate, chloride, sulfur dioxide, chlorine, ammonia, and ozone in the atmosphere is indicated.

Analysis of Water Samples

Samples of distillate from the water recovery unit used in the simulated flight beginning August 15 were obtained on the 19th and 26th of August. A composite urine sample was also obtained on the 26th of August. Analytical results are given in Table XII. Results obtained on a "normal" urine sample are included for comparison. The high volatiles content of the composite urine sample probably arose from bacterial action, since the sample was stored at room temperature.

TABLE VIII. IMPURITIES IN SPACE CABIN SIMULATOR IN TWO-MAN, 17-DAY TEST STARTED
ON 6/5/61 WITH 180 MM Hg OF O₂ AND 190 MM TOTAL PRESSURE

Compounds Measured	Amount Present at Hours (mg/m ³ at 25°C and 745 mm)				
	24	96	192	264	360
Acetylene	0.19	0.003	-	0.009	0.012
Unknown	-	0.004	-	0.020	0.017
Freon-22	0.012	0.01	-	0.037	0.028
Unknown	0.027	0.048	-	0.069	0.066
Unknown	0.0025	0.0037	-	0.012	0.0074
Ethylene Oxide	0.0017	0.0017	-	0.0087	0.0035
Acetaldehyde	0.11	0.11	-	0.18	0.19
Isoprene	0.040	0.030	-	0.051	0.051
Dimethyl Sulfide	0.012	0.012	-	0.012	0.012
Propionaldehyde	-	0.011	-	0.0036	0.0036
Acetone	0.89	0.74	0.67	0.64	0.56
Methyl Ethyl Ketone (Tentative)	0.059	0.012	-	0.041	0.012
Methanol	0.31	0.22	0.27	0.41	0.23
Ethanol	2.7	3.3	6.1	6.1	5.3
Methyl Propyl Ketone (Tentative)	0.40	0.16	0.032	0.11	0.048
Methyl Isobutyl Ketone	0.22	0.11	0.13	0.29	0.31
Toluene	0.78	-	-	0.13	-
Carbon Tetrachloride	0.026	-	0.0087	0.026	0.035

TABLE IX. IMPURITIES IN SPACE CABIN SIMULATOR IN TWO-MAN, 17-DAY TEST STARTED
ON 8/8/61 WITH 180 MM Hg OF O₂ AND 190 MM TOTAL PRESSURE

Compounds Measured	Amount Present at Hours (mg/m ³ at 25°C and 745 mm)					
	24	96	192	264	360	384
Unknown	0	0.003	0	0	0.014	0.005
Unknown	0.002	0.005	0.002	0.005	0.003	0.004
Ethylene Oxide	0	0.005	0.005	0.004	0.001	0.003
Acetaldehyde	0.037	0.079	0.063	0.061	0.051	0.067
Isoprene	0.023	0.030	0.029	0.020	0.020	0.023
Unknown	0	0	0	0.0025	0	0
Dimethyl Sulfide	0.005	0.006	0.005	0.01	0.007	0.007
Propionaldehyde	0	0.007	0.002	0.01	0	0.005
Acetone	0.22	0.22	0.11	0.45	0.08	0.11
Isobutyraldehyde (Tentative)	0	0	0	0.006	0	0
Methyl Ethyl Ketone	0.010	0.008	0.008	0.008	0	0.010
Carbon Tetrachloride	0.022	0.022	0.014	0.008	0.014	0.014
Methanol	0.053	0.13	0.030	0.040	0	0.050
Benzene	0	0	0	0.023	0	0
Ethanol	0.42	0.65	0.21	0.31	0.077	0.040
Methyl Isobutyl Ketone	0.095	0.095	0.051	0.044	0.022	0.037

TABLE X. INORGANIC CONTAMINANTS PRESENT IN CABIN AIR
IN A TWO-MAN RUN AT ATMOSPHERIC PRESSURE STARTED
ON 9/18/61

<u>Day No.</u>	<u>Contaminant (mg/m³ at 25°C and 745 mm)</u>					
	<u>SO₄⁼</u>	<u>Cl⁻</u>	<u>Cl₂</u>	<u>SO₂</u>	<u>NH₃</u>	<u>O₃</u>
1	1.24	0.81	0.006	0.024	0.82	-
3	1.24	0.39	-	0.024	0.097	-
4	1.86	0.62	0.058	0.148	0.097	0.008
8	1.67	0.40	0.029	0.008	0.119	0
10	1.68	1.80	0.029	0.008	0.169	0.018
11	1.59	0.50	0	0.042	1.86	0.004
14	1.95	0.60	0.029	0.026	0.162	0.025
15	1.77	0.53	0.029	0.008	0.198	0.022

TABLE XI. INORGANIC CONTAMINANTS PRESENT IN CABIN AIR
IN TEST RUN AT 190 MM PRESSURE AND 100% O₂. NO SMOKING
WAS ALLOWED. TEST STARTED ON 10/30/61

<u>Day No.</u>	<u>Contaminant (mg/m³ at 25°C and 745 mm)</u>				
	<u>SO₄⁼</u>	<u>Cl⁻</u>	<u>Cl₂</u>	<u>SO₂</u>	<u>NH₃</u>
0	0.27	0.12	-	-	0.211
1	0.27	0.04	0.029	-	0.26
3	0.27	0.09	0.029	0.024	0.063
7	0.39	0.12	0.029	0.003	0.338
8	0.27	0.09	0.088	0.003	0.063
10	0.27	0.12	0.029	0.008	0.204
14	0.27	0.12	0.029	0.003	0.204
15	0.27	0.04	0.029	0.024	0.26
16	0.39	0.12	0.029	0.003	0.338

TABLE XII. ANALYSIS OF WATER FROM DISTILLATION RECOVERY UNIT

Date	Sample	(mg/m ³)			
		Acetaldehyde	Acetone	Methyl Ethyl Ketone	Methyl Ethyl Alcohol
8/18/60/B	Distillate	.33	.76	.08	29.2
8/26/60/A	Distillate	.05	19.2	.5	60.0
8/26/60/B	Composite Urine	2.8	30.4	.8	56.0
"Normal" Urine ^(a)		.05	7.7	.1	14.0
					.6

^(a) Southwest Research Institute data. Actual concentrations will vary, but figures given are representative values.

The water was obtained by distillation of a mixture of urine and wash water, so the results are difficult to interpret. The high ethanol content of the distillate may arise from use of this substance by the test personnel for washing. As would be expected, volatile constituents are not separated from the water during distillation and appear in the recovered distillate. The distillate of August 26 was unacceptable for drinking purposes.

CONCLUSIONS

1. Trace components of metabolic origin which appeared in the atmosphere of a simulated space capsule during a series of experiments of 17 and 30 days duration include methanol, ethanol, acetaldehyde, acetone, methyl ethyl ketone, and others.
2. Inorganic materials found in air samples include sulfate ion, chloride ion, chlorine, sulfur dioxide, ammonia, and ozone.
3. Contaminants introduced with equipment or chemicals include methylene chloride, Freon-11 and Freon-12.
4. Abnormally high levels of ethanol and acetone were observed occasionally. This was probably associated with the use of these solvents in the space cabin.
5. A number of unidentified peaks appeared on a chromatogram obtained on a sample taken at a time when there was a malfunction of a waste disposal unit.
6. Contaminants in the cabin atmosphere can be kept at an acceptably low level by circulating the air once each minute through a purifier containing 40 - 50 pounds of activated charcoal.
7. The volatiles content of urine and recovered water taken from the cabin was abnormally high, due apparently to the fact that distillation did not separate volatiles formed during storage by bacterial action.

8. The data do not provide any evidence of atmospheric reactions, even though some of the impurities measured are highly reactive at higher concentrations. Throughout the runs, the atmospheric concentrations of both organic and inorganic contaminants fluctuated with changes in the operation of the simulator but did not appear to show any consistent trends that would indicate the gradual formation or depletion of any contaminants by means of atmospheric reactions.

RECOMMENDATIONS FOR FUTURE WORK

Air purification appears to be satisfactory for the missions which are presently being undertaken. When longer missions are undertaken in the future, however, the problem of trace atmospheric contaminants is expected to assume greater proportions. The results obtained in this investigation provide ample evidence that contaminants can occur in an enclosed system from a variety of causes and give some indication of the overall nature of the problem. Based on this investigation, the following recommendations are made as a guide to future planning in the design and operation of space capsules and simulators.

Improvements in Techniques for Sample Collection and Analysis

In the method presently being used for taking samples from simulated space cabins, the air is passed through a trap containing Ascarite to remove carbon dioxide. This is necessary because the system presently being used to remove water and carbon dioxide is capable of handling only 20 ml of CO_2 , while as much as 600 ml was found to be present in a 20-liter air sample. A Baralyme trap is used in the air circulation system of the simulator which might be expected to remove the same types of compounds as the Ascarite trap. Nevertheless, it is possible that the trap used during sample collection might remove additional trace components present in the cabin atmosphere and thus result in low results.

It might be possible to eliminate this difficulty by use of an enrichment column in combination with a flame ionization detector for the analysis. This technique is widely used in studies of the

aromas of foods and beverages. Instead of freezing out trace components with liquid oxygen, the trap is filled with the same column packing used to separate the atmospheric contaminants. Thus, it becomes a portable extension of the analytical column which can be transported separately to the site of sampling. The sample is collected by passing a large volume of air through it at room temperature or preferably lower. The use of a low temperature does not imply that the trace components are being condensed. It merely serves to retard their passage through the column and reduce prefractionation. Generally, a temperature 50° to 100°C lower than that used for chromatography will suffice. When the precolumn is connected to the analytical column, it should be positioned so that the gas flow is reversed from that used for sample collection to compensate for prefractionation during sample collection. With additional development work, conditions could likely be found which would permit collection of volatiles from the atmosphere free of carbon dioxide. The flame detector does not respond to water (or carbon dioxide), so it would be possible to "see" trace components even though they were eluted with some water. Also, the high sensitivity of the flame detector would make it possible to use much smaller air samples and so simplify the collection system without losing sensitivity.

Additional Analytical Work

Results obtained with the Space Cabin Simulator at the School of Aerospace Medicine indicate that organic contaminants in the atmosphere do not build up to a dangerous level in a period of 670 hours if the air is recycled continuously through activated carbon and Baralyme. However, this space environment is not complete in that it does not contain a photosynthetic gas exchanger or a system for the biological oxidation of organic wastes. Therefore, additional studies should be made on the nature of the volatile compounds emitted from cultures of algae being used for the generation of oxygen and food, and from symbiotic mixtures of algae and bacteria being used for the digestion of organic wastes.

Additional areas in which analytical results are incomplete include studies on the compounds emitted from human wastes and odors emanating from the preparation of foods.

Qualitative and quantitative studies such as those described previously should help to answer the following questions:

- (1) At what rate are organic compounds released into the atmosphere per man per day?
- (2) Is activated charcoal a suitable adsorbant for removal of all the contaminants which are likely to build up in the atmosphere a self-regenerating environment?
- (3) How much adsorbant must be used per man per day to keep contaminants at an acceptably low level?

It is believed that answers to these questions would contribute measurably to solving the problem of how to support life in a closed environment with the least amount of equipment.

Studies on the Purification of Air

In the simulator currently being used at Brooks Air Force Base, approximately 50 pounds of activated charcoal is provided for air purification for two men during simulated flights of 17 to 30 days. Undoubtedly, this would have to be increased for a larger crew and longer flights. Therefore, to cut down on weight as much as possible, an air purification system which can be regenerated should be developed.

Among other requirements, engineering studies of desorption would be needed to develop the best methods for use in a space environment. The use of treated charcoal might also have certain advantages, since certain treating agents have the ability to decompose impurities following adsorption, and thus increase the time interval before desorption or regeneration is required.

The simplest scheme would be to use two purifiers in parallel, arranged like large gas chromatographic columns. While one was in use, the other could be regenerated. The purifiers would be packed with a material from which bound contaminants could be released quantitatively, preferably by a simple process such as heating. The adsorbant should have a high holding capacity at ambient temperatures. Presumably, a solid such as activated charcoal would be used, but a liquid coated on a solid such as is used in gas-liquid chromatography cannot be counted out, since these materials have unique properties.

For example, it is possible to prepare a free flowing powder containing as much as 50% of a high molecular weight organic liquid. Materials could be chosen which have a particularly high affinity for the organic compounds present in the air and a low affinity for water and carbon dioxide. Liquid packings also have an advantage in that most organic compounds can be eluted from them quantitatively merely by raising the temperature.

The organic compounds that are eluted from the purifier could be disposed of by (1) collection in a cold trap and venting to space, (2) biological oxidation, or (3) combustion. Of these alternates, combustion seems the most desirable, since all of the organic compounds currently identified in the cabin simulator would be converted to carbon dioxide and water which could be fed directly into a photosynthetic unit. The simplest way to oxidize these compounds would be to pass the eluate from the purifier through a heated tube containing a catalyst such as Hopcalite. The use-regeneration cycle would be kept as short as practicable to minimize the amount of adsorbent that would have to be carried.

Air Monitoring Equipment for Space Capsules

The value of gas chromatography as a means of monitoring the air in a space cabin has already been demonstrated. During only two tests with the Brooks School of Aerospace Medicine Simulator, the following cases of atmospheric contamination were detected: (1) build-up of trace components due to malfunction of a waste disposal unit, (2) the presence of methylene chloride liberated from a plastic fabricated water recovery still, (3) a high concentration of acetone due to the fact that a bottle of it was opened to repair a Plexiglas window, and (4) air contamination with Freon-11 and Freon-12 resulting from use of an aerosol. All of these were the result of known factors in the operation of the simulator, but their detection at levels far below any toxic threshold illustrates the value of atmospheric monitoring. Therefore, it is obvious that gas chromatography would be of great value in monitoring the air in a space cabin to detect potential hazards before they become serious. It has several great advantages which would be difficult to duplicate with any other type of instrumentation. These are (1) very high sensitivity, (2) capacity for identifying trace components in the air by retention times, (3) general applicability - it can

be used to measure oxygen, carbon dioxide, water, carbon monoxide, helium, etc., in the air as well as trace components, (4) speed, and (5) simple and rugged instrumentation. It should be possible to construct a miniaturized gas chromatograph that would permit complete analysis of the air in a space cabin in from 0.5 to 5 minutes. Moreover, chromatographic apparatus may be developed that would monitor the air in a capsule continuously and give a warning signal in response to a change in the oxygen or carbon dioxide content of the air. Thus, a single instrument of comparatively simple design could be used to replace more complex and less versatile infrared analyzers, etc., for monitoring the air in a space capsule.

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